HIGH TEMPERATURE THERMOCOUPLE
RESEARCH AND DEVELOPMENT PROGRAM

MONTHLY PROGRESS REPORT NUMBER 11
Period 1 April 1964 to 1 May 1964
Contract Number NAS 8-5438
Request Number TP 3-83547

prepared for
GEORGE C. MARSHALL SPACE FLIGHT CENTER
Huntsville, Alabama

R

work performed by
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Date of Publication: 10 May 1964

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ABSTRACT

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This report covers the period 1 March 1964 to 1 April 1964, under Contract NAS 8-5438, which calls for twelve months of research and development of a high temperature thermocouple capable of measuring rocket engine exhaust temperatures in the 3000°C range, under adverse conditions of oxidation, erosion, vibration and shock.

The primary objectives of the program are to advance the state-of-the art of high temperature thermometry and to develop an end product suitable for in-flight temperature measurements on the SATURN vehicle.

Calibrations were continued into the over 4000°F range. Investigations of high temperature oxidation resistant coatings were continued. Changes were withheld pending receipt of results of NASA investigations which were not, at the time of this report, completed. Materials tests, regarding notch sensitivity and ductlity of the sheath material, were made. The contract is scheduled for termination on 17 June 1964. Therefore work has started on preparation of the final engineering report.

Author

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SECTION I

SUMMARY

1.0 Period Covered

This report covers the period 1 April 1964 to 1 May 1964.

1.1 Statement of Work

The Contractor shall advance the state-of-the-art of high temperature thermometry and specifically improve the technique of accurately measuring high temperatures by designing, fabricating, testing, and delivering nine (9) thermocouple probes capable of operation in the 3000°C temperature range under adverse conditions of erosion oxidations and high stress levels for useful period of time. Also, present methods of thermocouple probe fabrication will be modified such that the end product will be suitable for in-flight temperature measurements on the SATURN vehicle.

To accomplish the above objectives the Contractor shall consider and explore specific R & D efforts as follows:

a. Develop of the physical structure of an immersed probe to attain minimum drag and highest resistance to bending and shear forces.

b. Ascertain the best combination of ingredients in the protective coating of the probe to extend the term of oxidation resistance.

c. Determine the best combination of compensated lead wires for use with the immersion type probes.

d. Incorporate latest state-of-the-art materials as potting and sealing elements in the base of the probe.

e. Determine effects of reactions between oxide coatings and tungsten in relation to the emf output.

f. Establishment of rates of erosion for different types of refractory coatings such as tungsten disilicide, carbides and cermets when subjected to high velocity, high temperature gas streams.

1.2 Progress

a. Calibrations

One gauge was subjected to a total of three calibration runs from about 2000°F to 5000°F. Results were in excellent agreement with data taken by Hoskins, Aerojet and others, as well as with previous runs made by ACL.

b. Protective Coatings

Some encouragement in the use of thin oxide coatings has resulted from recent work.

c. Ductility & Notch Sensitivity

Recent work on surface treatment of the material to reduce notch sensitivity has resulted in an increase in cold strength by about 50%. The brittle to ductile transition temperature of the sheath material has been established at about 400°C.

SECTION II

PAST PROGRESS

2.0 General

Previous effort was reported in ACL Progress Reports T-1097-1 through T-1097-10.

2.1 Prototype Design and Development

As was previously reported, objectives for the first prototypes were limited to the 4000°F - 4500°F range in the interest of accumulating test data for analysis, the results to be utilized in future design.

A design approach for the prototype gauges was selected, and drawings prepared, detailing means of fabrication and assembly.

Investigations made into fabrication techniques involved in working vapor deposited Tungsten, resulted in improved material handling techniques.

Shock and vibration tests, performed on a prototype mock-up, resulted in a conclusion that the sheath material was intrinsically capable of withstanding the specified shock and vibration requirements.

Samples of various types of compensation lead wires were ordered for test and evaluation.

An evaluation of the SRI calibration tests for ACL Type 4734 gauges was made, resulting in a conclusion that an optimum immersion depth might be in the order of 1 - 1/2 inches in an isothermal region.

The two Type 4734 gauges tested by N.A.S.A., and returned to ACL were examined and results of the examination were reported.

A test of a "no-insulation" approach was started, but was aborted due to a failure in the test oven. Such tests were subsequently continued.

2.1 Prototype Design and Development (Continued)

Three prototype gauges were delivered to M-ASTR-I, on 17 October 1963, for test and evaluation. Calibration of this type of gauge indicated a shift in emf output to a higher value than that shown in previous calibrations. The shift was believed due to a spurious emf contributed by the "compensated" lead wires. The curves, however, paralleled the curves taken by Southern Research Institute, as well as those predicted by ACL.

Further tests verified the presence of lead wire errors.

Analyses of form and shock drag loads were made. The results will be considered in future design.

Investigations of oxidation resistant coatings were continued. Accumulated data was reviewed, and tabulated for comparison and reference.

Response test performed on one Type 4735 gauge yielded response as low as 45 milliseconds from ambient air to boiling water. Lead wire tests resulted in a conclusion that the thermocouple materials should be used in lead extensions for best accuracy. Further investigations of oxidation resistant coatings, and insulators verified the conclusion reached in earlier tests. Design of the second generation gauges was continued.

Three second generation gauges were delivered to M-ASTR-I on 26 February 1964 for test and evaluation. These gauges incorporated thermocouple materials as lead wire, and elimination of BEO insulation in the Hot Zone. Calibrations of this type of gauge showed an increase in the upper temperature limit, and virtual elimination of lead wire error. emf output curves essentially tracked predicted values.

Continuation of calibration tests resulted in verification of calibration curves previously developed.

Further calibration and stability tests were highly repeatible. A positive drift, diminishing in extent with cycling, was noted.

SECTION III

CURRENT PROGRESS

3.0 General

ACL has directed effort in the current reporting period toward verifying calibrations at temperatures in the 4000°F to 5000°F temperature range, with longer periods of time spent at stabilized cavity temperatures, than in previous calibrations.

ACL had anticipated receipt of data from the tests planned by M-ASTR-I at Southern Research Institute prior to the date of this report. Such information was planned for use in making any indicated changes in the Type 4735 gages. It has not, apparently been possible to complete the SRI tests. As a consequence, ACL plans to proceed with fabrication of the three final gages on the basis of ACL tests. If the SRI data is made available prior to conclusion of work under this contract on 17 June 1964, in sufficient time, ACL plans to incorporate any indicated changes.

3.1 Progress

3.1.1 Calibrations

One type 4700 gage was subjected to tests at temperatures from about 2000°F to about 4200°F. Fewer data points were taken over the range to allow more time for stability at each point observed with the optical pyrometer. The temperature vs emf data were plotted and are shown in Figure 1. The Comparison plot for agreement with the published Hoskins data is shown in Figure 2. Since no published data was available at the time of the tests for the temperature range from 4200°F up, the local Hoskins representative was contacted to see whether any unpublished data was available. The Hoskins representative reported that he had been advised by his engineering staff that the curve could be extrapolated from the 4200°F point to 5000°F without an appreciable loss of accuracy. Plots of millivolts per degree F in this range do not agree with such extrapolations, however, nor do they agree with either the Englehard curve or previously taken ACL data. Therefore they were not used.

3.1.1.1 Test Method

The test methods employed in these calibrations were identical in every essential detail with those described in the last report.

3.1.1.2 Run No. 1

Run No. 1 was made from 2093°F to 4217°F and return to 2257°F. Total running time was 3 hrs. 40 min., with about 1 hr. at 4217°F. After cool-down, the gage was examined. No evidence of adverse effects were observed. The output curve from the ascending portion of the run matched the Hoskins curve, point for point, over nearly the entire range. Any deviations were within the experimental error limits. A positive drift was observed during the descending run.

3.1.1.3 Run No. 2

Run No. 2 was made from 2138°F to 4082°F. No descending run was made. Total running time was 2 hrs. 20 min, with about 1 hr. above 4000°F. No adverse effects were observed as a result of this run. The positive drift observed during the descending portion of Run No. 1 had apparently stopped.

3.1.1.4 Run No. 3

Run No. 3 was made from 2241°F to 4136°F. No descending run was made. Total running time was 2 hrs. 20 min., with about 1 hr. above 4000°F. Excellent agreement was seen between Run No. 2 and Run No. 3. The positive drift was still believed to be present, but had evidently diminished to the extent that its presence was difficult to detect, when pyrometer operator error and the limits of accuracy of the measuring apparatus are taken into account.

3.1.2 Low Temperature Calibrations

Very little information has been found in the literature concerning the output of Tungsten-Rhenium thermocouples at low temperatures. ACL, therefore, performed calibrations on a test thermocouple at three temperatures: The boiling

3.1.2 Low Temperature Calibrations- Continued

point of Liquid Nitrogen (-320 F), the equilibrium temperature of a mixture of solid CO2 and Acetone, and the melting point of ice.

The test gage was fabricated from Tungsten vs Tungsten 26% Rhenium wire, .020 inch diameter, Hoskins Tungsten Lot No. 34, and Tungsten 26% Rhenium Lot No. 2610. The lowest output in the calibration curve provided for this matched set of wires was .292 mv at 200°F.

Copper leads attached to each of the thermocouple wires were brought out to a Type 8662 L & N Precision Portable Potentiometer, where the output was read, after the bridge was compensated.

The hot junction of the test gage was first immersed in the ice bath, with the positive lead of the gage attached to the positive terminal of the bridge. There was no discernible output. The leads were reversed, with the same result.

The hot junction was then immersed in the acetone and CO2 bath, and the same results were obtained. In the LN2, the same result was again observed.

The junction was then placed in the ambient air, and the transition between the copper and the thermocouple materials was immersed in the LN2. A negative output of .03 millivolts was measured on the bridge. The same test performed in the acetone and CO2 yielded a negative output of .005 mv. In the ice bath, there was no discernible output, either negative or positive.

The effects as measured above are of particular interest in applications where the transition section of the gage might be maintained at some low temperature, while the hot junction was operating at an elevated temperature. The tests indicate that Tungsten vs Tungsten 26 Rhenium has a negligible output at low temperatures, and that errors in the output are very low if copper lead extensions are used when the transition is maintained at temperatures from -320 F to near ambient. This opens up the possibility of dispensing with a reference junction while still maintaining accuracy within acceptable limits.

3.1.3 Vibration Test

Vibration testing had been performed early in the program on a mock-up of the Type 4735 Gauge. A completed gage had not, however, been subjected to vibration tests. One second generation gauge, therefore, was subjected to investigative tests to determine its response to sinusoidal vibratory inputs in the range 5 to 2000 cps.

The gage tested had been temperature cycled repeatedly from ambient to more than 5000°F. Any effect of recrystallization on the physical structure of the sheath had, therefore, been fully realized. The principal concern in this regard was whether high temperature cycling would induce an early failure in the sheath material, since any gage calibrated to the temperatures of interest would have had every opportunity to recrystallize, and it is hardly likely that a virgin gage would be used in an application.

During the tests, both input to the fixture and output at the gage nut were monitored with calibrated accelerometers, over the frequency range and inputs shown in the data sheet of Figure 3. The gage was vibrated at each resonant frequency for two minutes. The resonance search was conducted over the range with a logarithmic sweep in 45 minutes.

The gage was examined following the test. No evidence of structural damage was seen. The gage function was unimpaired as a result of the test.

The input was held to 14g at the resonances for two reasons: to permit measuring the Q of the gage, and to prevent extreme amplification at the resonant points. Table 1, below, lists resonant points. Table 1, below, lists resonant frequencies and the amplification factor for each.

Table 1

AMPLIFICATION FACTOR Q

	Resonant Frequency cps	Input g	Output g	Amp. Factor Q
Z-Axis	125	14	24	1.7
	270	14	24	1.7
	450	14	50	3.6

Table 1 (Continued)

AMPLIFICATION FACTOR Q

	Resonant Frequency cps	Input g	Output g	Amp. Factor Q
	700	14	36	2.6
	1310	14	60	4.3
X-Axis	440	14	20	1.4
	560	14	50	3.6
Y-Axis	105	14	28	2.0
	155	14	22	1.6
	475	14	42	3.0
	510	14	47	3.4
	1125	14	44	3.1
	1400	14	58	4.1

The "tube" referred to in the data sheet is the sheathed extension lead wire of about 18 inches in length, with a B-nut on the end. The reaction of this lead may have contributed to the relatively high response as seen above.

3.1.4 Materials Test

Because of concern with the strength and ductility of the sheath material used in the Type 4735 gages, an attempt has been made to ascertain several characteristics over the temperature range from ambient to the brittle-to-ductile transition temperature. It is well established that Tungsten is a brittle material at normal temperatures. A general consensus seemed to be that vapor deposited Tungsten did not develop ductility at any temperature, and that vabor deposited Tungsten tubing exhibited little or no room temperature strength. ACL has long been convinced that this is not true because of the shock and vibration tests run very early in this project. A serious concern has existed, however, as to whether the sheath materials could be surface finished without serious loss of strength, at room temperatures.

3.1.4 Materials Test (Continued)

The brittle-to-ductile transition temperature has been established as approximately 400 C.

Four Tungsten tubes, each 3/8 inch long, .250 inch inside diameter, and .030 wall thickness were prepared for the test. Two of the tubes were surface ground on a belt-sander to remove the rough, as-received surface. Approximately .005 inch of material was removed in the grinding. The specimens were then placed horizontally between the platens of a compression tester and loaded radially until failure occurred. Failure was defined as occurring when the tube collapsed. Table 2, below, shows the results of this test.

Table 2

COMPRESSION TEST

Sample Surface Character	Pounds Loading at Failure
Ground	150
Unground	95
Ground	140
Unground	105

One of the points of concern had been that grinding would provide a detrimental condition, in that it would create small surface marks that might aggravate the known notch-sensitivity of the Tungsten. The results of the test indicate that surface grinding actually improved the strength of the material, under the conditions of test, by about 50%. The nature of the fractures in the material at failure support the argument that the grinding removed very sharply defined microscopic peaks and valleys in the surface of the material, thus eliminating incipient points of failure due to stress concentration. The unground specimen both broke axially, with long cleavages. The ground specimens suffered a nearly explosive type of fracture with very small pieces resulting from the fracture, and developed no well-defined pattern of cleavage. It would appear, then, that grinding the surface permits higher loading by permitting the locked-in stresses to build to a higher value, with cleavage along a random pattern of grain boundaries when the stresses are relieved at failure.

TABLE NO. 3

CALIBRATION DATA - TYPE 4700 GAGE
RUNS 1, 2, AND 3

Temp. °C	Temp. °F	Output MV	Time
Run No. 1			
1145	2093	16.582	2:20
1430	2606	23.666	2:40
1651	3004	27.515	3:00
1990	3614	34.367	3:20
2325	4217	38.856	3:40
2325	4217	38.111	4:40
2019	3708	33.531	5:00
1731	3148	27.780	5:20
1517	2763	23.250	5:40
1236	2257	16.649	6:00
Run No. 2			
1170	2138	14.952	10:40
1401	2554	20.455	11:00
1626	2959	25.436	11:20
1938	3520	31.675	11:40
2236	4057	36.706	12:00
2250	4082	36.880	1:00
Run No. 3			
1227	2241	16.452	2:55
1515	2759	23.362	3:15
1643	2991	26.215	3:35
1951	3544	32.264	3:55
2236	4057	37.240	4:15
2280	4136	37.438	5:15

A/L THERMOCOUPLE, TPR 4735

4-24-64

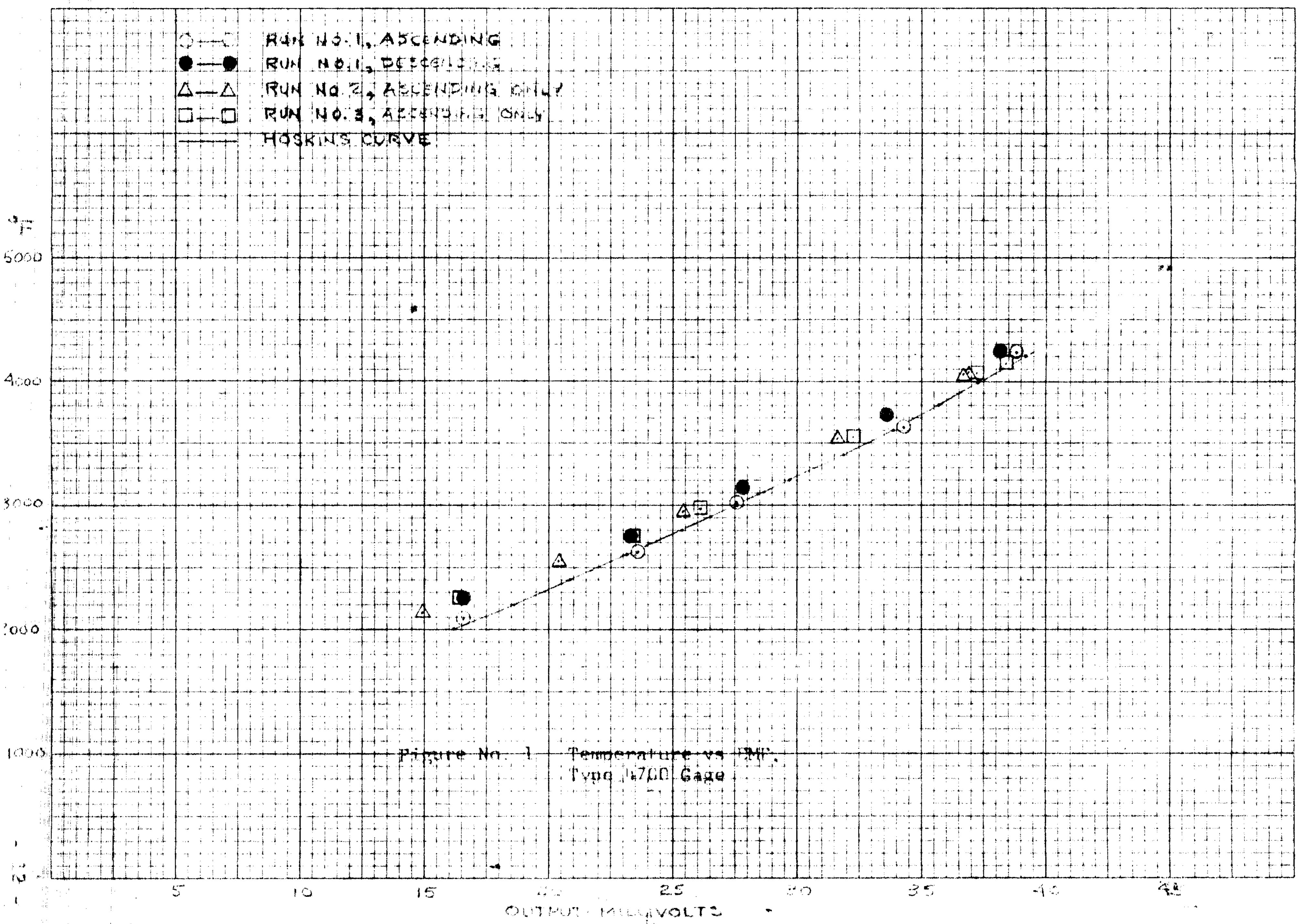

Figure No. 1 Temperature vs EMF,
Type 4700 Gage

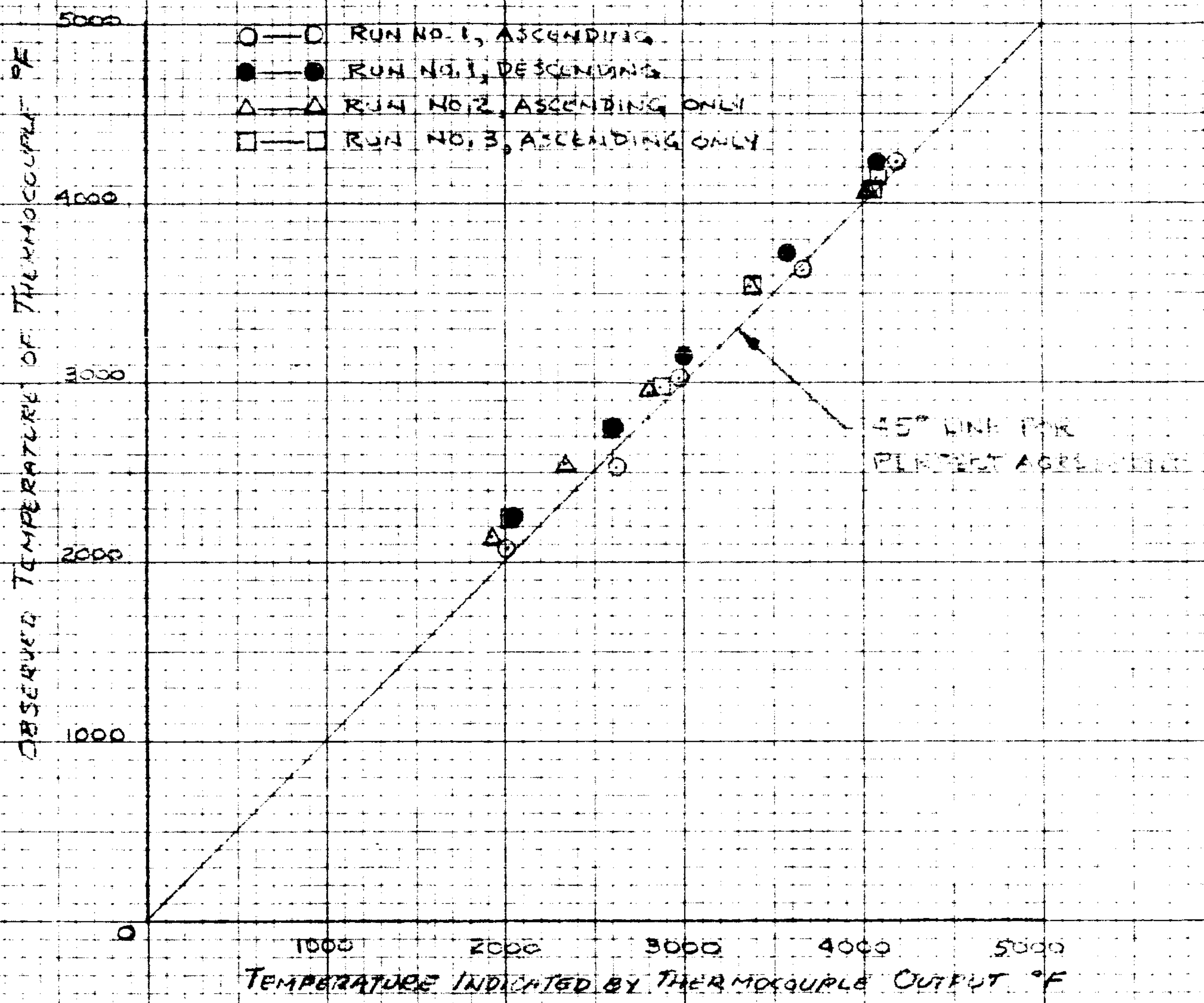

Figure No. 2 - Comparison of Observed Temperature and Indicated Temperature versus Hoskins Curve

AUTO-CONTROL LABORATORIES INC (ACL)

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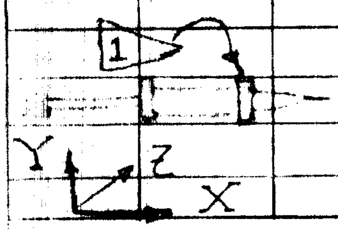
CUSTOMER ACL	PAGE 1 OF 1
PART DESCRIPTION THERMOCOUPLE	JOB NO. T-10[illegible]
PART NO. 47[illegible]-Z	DATE 4-13-[illegible]
SERIAL NO. 001	SPEC.
REF.	TEST BY [illegible]
	WITNESS [illegible]

VIBRATION DATA SHEET

AXIS	TEMPERATURE DEG. FAH.	RESONANCE SEARCH	RESONANT FREQUENCE (CPS)	FREQUENCY RANGE (CPS) FROM	TO	VIBRATION AT RESONANCE (MIN.)	VIBRATION CYCLING (HOURS)	ACCELERATION (G'S)	DISPLACEMENT (IN. P-P)
Z-Z	Rm	Yes	—	5	14				0.1
			—	14	45			4.0	
			—	45	8[illegible]				0.0[illegible]
		Yes	—	4[illegible]	2000			INPUT 14.0 OUTPUT —	
			125			2.0	Tube	24.0	
			270			2.0	Tube & Probe	24.0	
			450			2.0	Probe	50.0	
			700			2.0	Tube & Probe	36.0	
			1310			2.0	" "	14.0 60.0	
X-X	Rm	Yes		As shown above				As shown above in 1st four columns.	
			140			2.0	Probe	INPUT 14.0 OUTPUT 20	
			560			2.0	Tube	14.0 [illegible]	
Y-Y	Rm	Yes	—	As shown above				As shown above in 1st four columns.	
			105			2.0	Tube	INPUT 14.0 OUTPUT 28.0	
			155			2.0	Tube & Probe	22.0	
			475			2.0	Probe	42.0	
			510			2.0	Tube	47.0	
			1125			2.0	Tube & Probe	44.0	
			1400			2.0	"	14.0 58.0	



NOTES 1 Out Put Accelerometer Was Mounted At This Point.

SECTION IV

PROGRAM FOR NEXT INTERVAL

4.0 Objectives for the Interval 1 May 1964 to 1 June 1964 are:

a. Prepare for delivery of the final three gages on 17 June 1964.

b. Continue work on Final Engineering Report.

c. Review results of SRI tests, if available.

d. Continue search for oxidation resistant coatings.

SECTION V

STATEMENT OF MAN HOURS

5.0 Hours by Category

Category	Previous Periods	Current Periods	To Date
Engineering	794.5	37.0	831.5
Clerical	145.5	20.0	165.5
Fabrication	763.5	9.0	770.5
Consulting	20.5	- 0 -	20.5
Drafting	61.0	- 0 -	61.0